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AEC RESEARCH AND
DEVELOPMENT REPORT

54598

DEVELOPMENT OF HIGH STRENGTH COLUMBIUM AND TANTALUM ALLOY TUBING

THIRD QUARTERLY PROGRESS REPORT

JUNE 1, 1963 TO AUGUST 31, 1963

OCTOBER 11, 1963

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Westinghouse Electric Corporation

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(TID-4500, 20th Edition)

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June 1, 1963 - August 31, 1963

R. W. Buckman, Jr.
J. L. Godshall

Contract AT(30-1)3108

U. S. ATOMIC ENERGY COMMISSION

October 11, 1963

Westinghouse Electric Corporation
Astronuclear Laboratory
P. O. Box 10864
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Development of High Strength Columbium and Tantalum Alloy Tubing

Third Quarterly Progress Report

by

R. W. Buckman, Jr.
J. L. Godshall

ABSTRACT

The feasibility of drawing B-66 to tubing was demonstrated. A solid B-66 rod was gun-drilled and then reduced by drawing, 80%, to 0.5 inch O.D. x 0.062 inch wall tubing at 600°F. An attempt to draw B-66 to tubing at room temperature was unsuccessful because of a lubrication failure. This resulted in excessive galling between the die and tube O.D. from which a crack was initiated. Tube drawing is being done at the facilities of the Superior Tube Company.

Extrusion of 69 pounds of B-66 and 142 pounds of T-111 tube blanks at a reduction ratio of 7:1 was accomplished at the facilities of Nuclear Metals, Inc. The extrusion constant for the B-66 was 80-81.7 ksi and 76-85.5 ksi for the T-111. The results of the tube blank extrusions verified the data from the extrudability evaluation that were described in the Second Quarterly Progress Report.

The B-66 extrusions were conditioned and yielded 43.75 pounds of tube blanks, a 63% recovery of useable material based on the starting extrusion billet weight. The as-conditioned B-66 tube blanks were successfully warm tube reduced 55% from a 2 inch O.D. x 0.25 inch wall to a 1.375 inch O.D. x 0.156 inch wall.

end

The T-111 extrusions are being conditioned and will be annealed for one hour at 1500°C (2700°F) prior to tube reducing.

During the final quarterly period, the B-66 and T-111 will be processed to the required finish tubing sizes. After inspection, the tubing will be shipped to the designated laboratories for evaluation.

SUMMARY

This is the third quarterly progress report on AEC Contract AT(30-1)3108, "Development of High Strength Columbium and Tantalum Alloy Tubing". The results of the metallurgical examination of the solid rod and tube blank extrusions are presented. The feasibility of warm tube drawing B-66 is discussed and the results of warm tube reducing of the B-66 tube blanks are also presented.

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THIRD QUARTERLY PROGRESS REPORT ON DEVELOPMENT OF HIGH STRENGTH COLUMBIUM AND TANTALUM ALLOY TUBING

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I. INTRODUCTION

During the third quarterly period the evaluation of the Phase I extrusions was completed. The feasibility of producing B-66 tubing was demonstrated. A portion of extrusion B-66-1 was "gun-drilled", and then processed to 0.5 inch O.D. by 0.062 inch wall tubing by warm drawing (600°F). The warm tube drawing evaluation was conducted at the facility of the Superior Tube Company under the direction of Messrs. L. Shaheen and W. Hunsberger.

The Phase II B-66 and T-111 extrusion billets were successfully extruded to a nominal 2 inch O.D. x 0.25 inch wall tube blank. The extrusion was done at the facility of Nuclear Metals Inc. under the direction of Messrs. W. Toeffin and G. Clattenburg. The B-66 Cb tube blank extrusions were lathe conditioned and successfully warm tube reduced 55%. The T-111 Ta tube blank extrusions have been conditioned and are being annealed 1 hour at 1500°C prior to tube reducing.

II. PROGRAM STATUS

A. Phase I Extrusion Evaluation

The Phase I solid rod extrusions, which were used to establish the extrusion parameters for the B-66 and T-111 tube blanks, were examined metallographically. A summary of the metallurgical evaluation is contained in Table I. Increasing the extrusion Cb,Ta speed from 275 to 1000 inches per minute had no significant effect on the as-extruded hardness or 1 hour recrystallization temperature of B-66. The detrimental effect of the increased extrusion speed on surface degradation has been described previously(1). Figure 1a is a transverse section from the tail end of the extrusion B-66-1 and is representative of the as-extruded microstructure. Surface contamination could not be detected by metallographic examination. Cb,Ta Increasing

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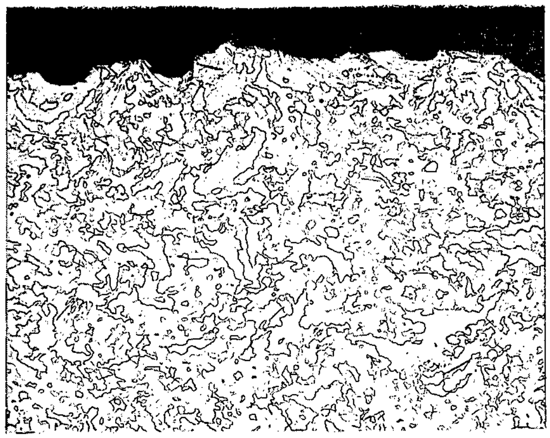
TABLE I Summary of Metallurgical Evaluation of Phase I Extrusions

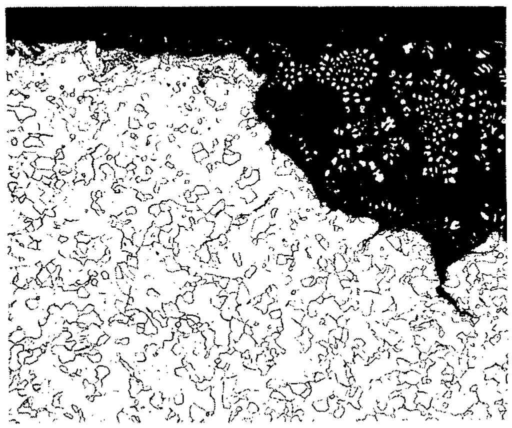
	Extrusion			
	B-66-1	B-66-2	T-111-1	T-111-2
Extrusion Temperature, (°F)	2200	2200	2200	2300
Extrusion Speed (inches per minute)	275	1000	175	1000
Extrusion Ratio	7:1	7:1	7:1	7:1
DPH Hardness[a] 10Kg Load				
As Extruded	240/228	235/229	285/287	281/281
1100 °C	234/230	230/227		
1200 °C	225/226	228/227		
1300 °C	216/228[b]	221/227[b]		
1400 °C	225/231	223/229	232/232	235/228
1500 °C			226/230[b]	226/227[b]
1600 °C			227/224	222/227

(a) First hardness value represents nose section and second value tail section. The annealing time was 1 hour.

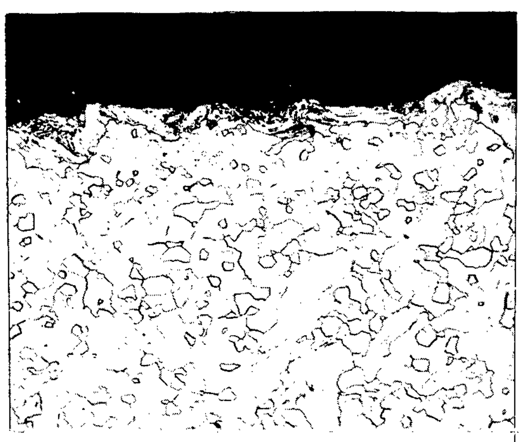
(b) Formation of recrystallized grains 100% complete.



a. B-66-1 Tail, 150X



b. B-66-2 Nose, 100X



c. B-66-2 Nose, 150X

Figure 1 - As-Extruded Microstructures of Phase I B-66,
Transverse Section
(Reduced approximately 40% in reproduction)

the extrusion speed of B-66 resulted in surface tearing, Figure 1b, and an apparent reaction of the steel cladding which resulted in a contaminated layer, Figure 1c. Contamination was less than 0.005 inch deep, thus showing the steel cladding afforded excellent protection to the B-66 billets during the extrusion operation.

Increasing the temperature and speed did not affect the as-extruded micro- Cb, Ta structure of the T-111 extrusions. The 2300°F extrusion temperature is below the recrystallization temperature of T-111. The "as-extruded" microstructures observed for both Phase I T-111 extrusions were similar and were typical of as-worked material. A representative microstructure taken from the nose section of extrusion T-111-1 is shown in Figure 2. As noted earlier the as-extruded T-111 had an excellent surface condition with no significant surface contamination.

The following projections were made for the Phase II tube blank extrusions based on the results of the Phase I metallurgical evaluation:

1. Minimal surface conditioning will be required.

2. No heat treatment will be required for the B-66 tube blanks prior to the initial tube reduction.

3. The T-111 extrusions will require annealing prior to tube reducing.

B. <u>Warm Drawing Evaluation</u>

The B-66-1 extrusion, after straightening by hammer forging at 950°C (1750°F) was lathe conditioned and centerless ground to a 0.963 inch diameter by 15-1/8 inch long rod. The microstructure of the as-straightened rod is shown in Figure 3a. The Vickers hardness was 269 DPH. After annealing for 1 hour at 1200°C (2200°F) the microstructure was more than 95% recrystallized. A hardness of 232 DPH indicated that annealed mechanical properties had been recovered after this treatment. The microstructure of the "as-annealed" rod is shown in Figure 3b.

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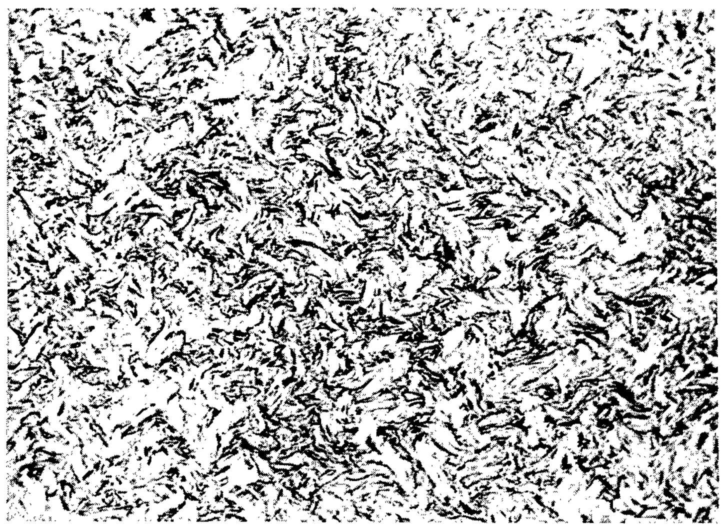
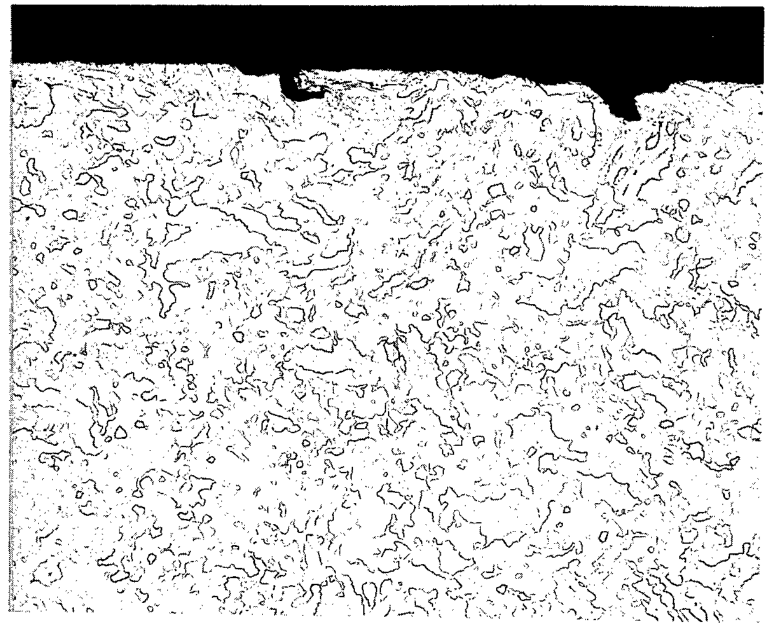
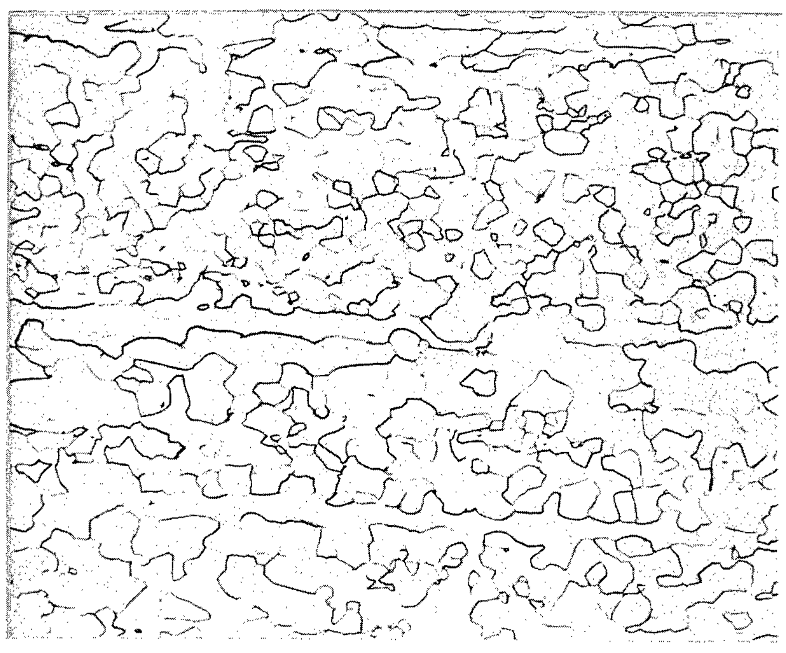


Figure 2 - "As-Extruded" Microstructure of T-111-2, Transverse Section, 150X



a. As-Straightened, 150X



b. After Annealing for 1 Hour @ 1200°C, 200X

Figure 3 - Microstructure of As-Straightened and As-Annealed B-66-1 Extrusion Used for Tube Drawing Evaluation

The annealed rod was "gun-drilled" at the Superior Tube Company. The finish machined size of the rod was 0.935 inch O.D. x 0.187 inch wall. It was planned to draw this tube to a finished size of 0.5 inch O.D. by 0.062 inch thick wall. The drawing was scheduled to be done at 600°F. A dry film lubricant, proprietary to the Superior Tube Company, was used for lubrication during the drawing operations. The schedule of reductions used for the warm tube drawing of B-66 is outlined in Table II. After the fourth drawing pass the tube was annealed for 1

Type → TABLE II Schedule of Warm Tube Drawing of B-66-1

	O.D. (Inches)	Wall Thick (Inches)	% Reduction of Area Per Pass	Total
Gun Drilled	0.935	0.187		
1st draw	0.875	0.160	18.0	18.0
2nd draw(a)	0.795	0.130	24.4	38.0
3rd draw	0.710	0.105	26.0	54.5
4th draw(b)	0.616	0.083	30.4	68.3
5th draw	0.549	0.062	31.7	78.4
6th draw	0.505	0.062	9.05	80.3

(a) Annealed 1 hour at 1100°C (2000°F) after 2nd draw.

(b) Annealed 1 hour at 1200°C (2200°F) after 4th draw.

hour at 1200°C. This treatment produced a 100% recrystallized microstructure with an ASTM grain size 8. The annealed tube was then cut in half and finish drawing at room temperature was attempted on one of the pieces. After 80% of the tube was through the die, the draw bar on the draw bench fractured. Upon regripping, a split developed near the die opening and propagated forward, thus, terminating the initial cold drawing evaluation of B-66. p. 8

The failed B-66 tube is shown in Figure 4. Examination of the failed tube indicated that during the room temperature drawing, a lubrication failure developed. As a result, excessive metal buildup occurred at the die entrance from galling action. The area of metal buildup and fracture initiation is shown in Figure 5. The progressive nature of the failure is evident. Figure 6 is a photomicrograph of a section taken immediately preceding the fracture. The crack propagating from the layer of metal buildup was arrested in the base metal.

The remaining half of the recrystallized tube was drawn at 600°F to a finish size of 0.5 inch O.D. x 0.062 inch wall x 18 inch long. A portion of this tubing is shown in Figure 7. Dye penetrant inspection did not show any O.D. surface defects, and ultrasonic and eddy current inspection failed to reveal any I.D. or internal defects that were greater than 10% of the wall thickness.

The tube drawing evaluation indicated the feasibility of drawing B-66 at room temperature if adequate lubrication can be provided, and that satisfactory B-66 tubing can be produced by warm drawing.

Weldability of the B-66 tubing produced under the warm tube drawing evaluation was evaluated by making a simulated butt weld using tungsten inert gas welding techniques. A 100% penetration was achieved. Visual examination showed the weld to be sound and free from defects. The weld is shown in Figure 8.

C. Phase II Extrusion

The Phase II T-111 starting billets were finish machined to the required configuration(2) and shipped to Nuclear Metals Inc. for extrusion. Data pertinent to the Phase II T-111 extrusion billets are given in Table III.

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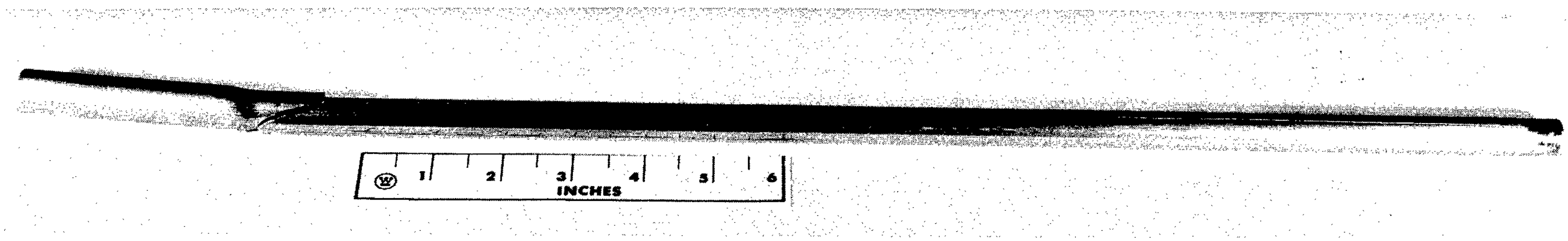

Figure 4 - B-66 Tube Which Failed During Room Temperature Drawing Pass

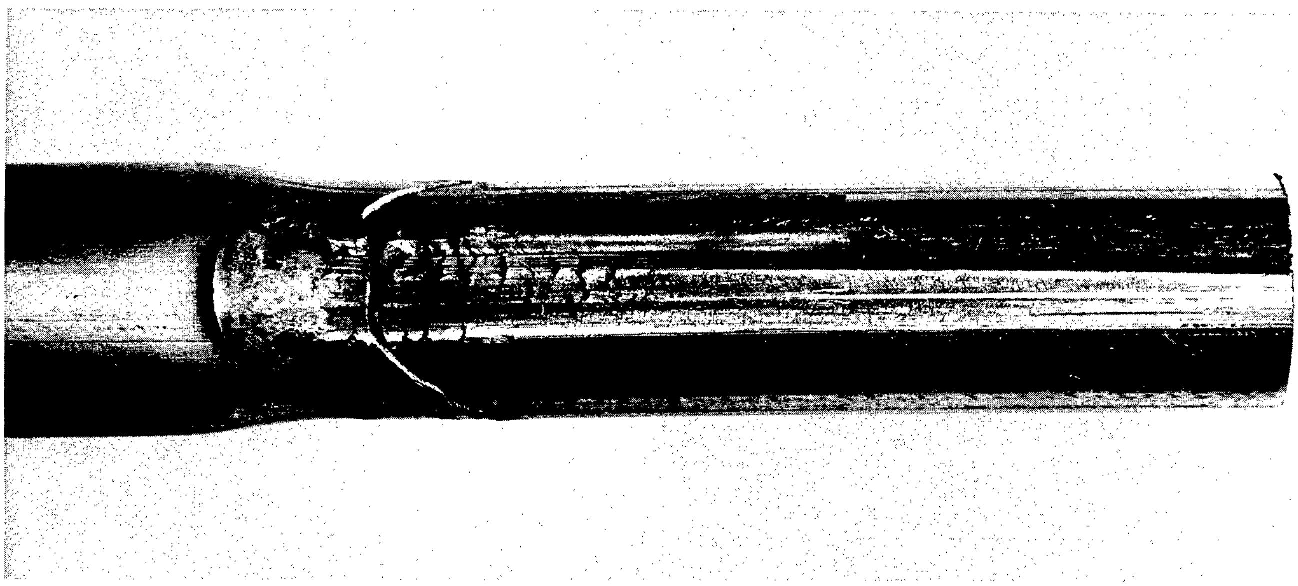

Figure 5 - Failure Area on B-66 Tube Drawn At Room Temperature, 2.75X

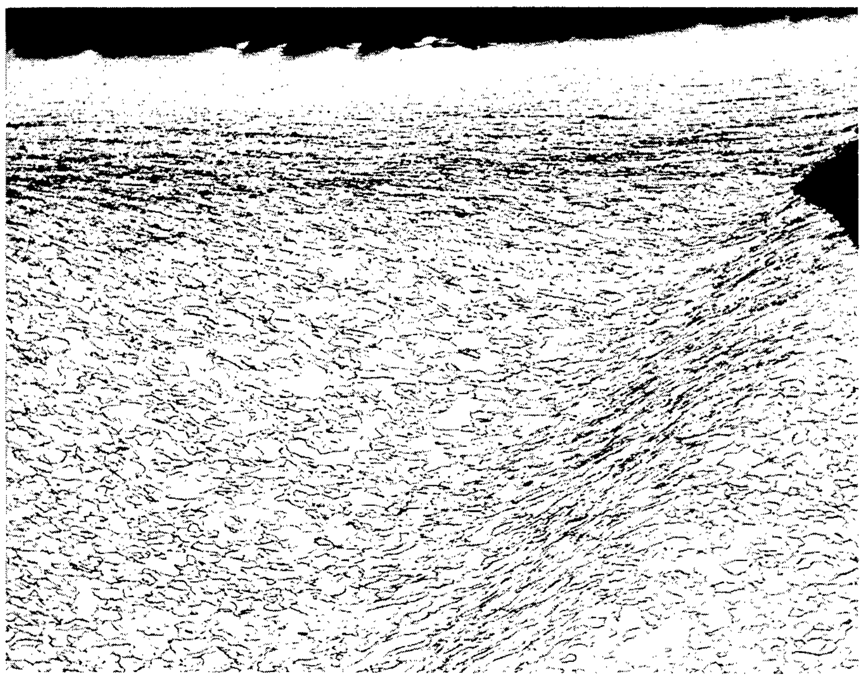

Figure 6 - Photomicrograph of Longitudinal Section From Area Preceding Failure During Room Temperature Drawing of B-66, 200X

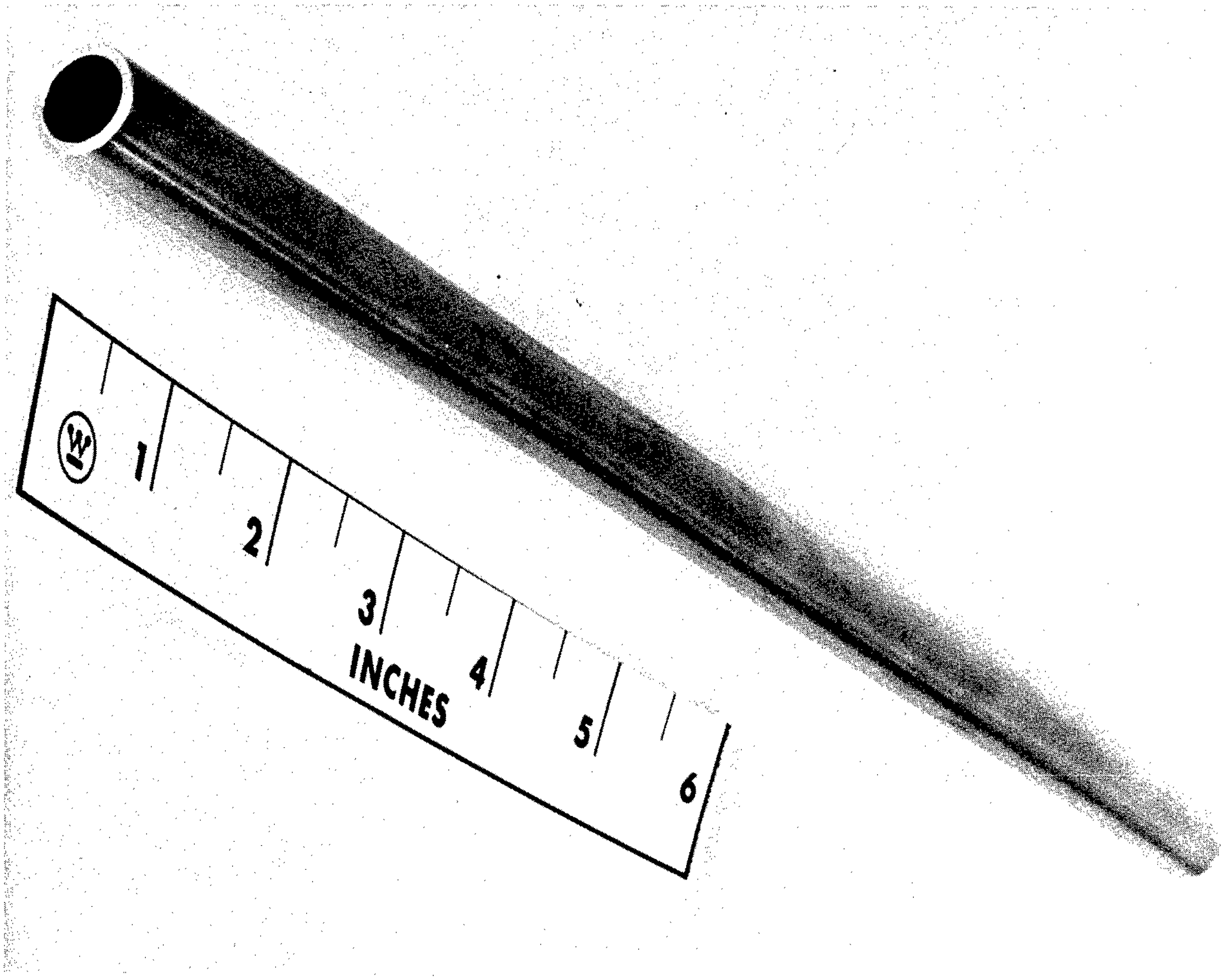

Figure 7 - B-66 Tubing As Warm Drawn

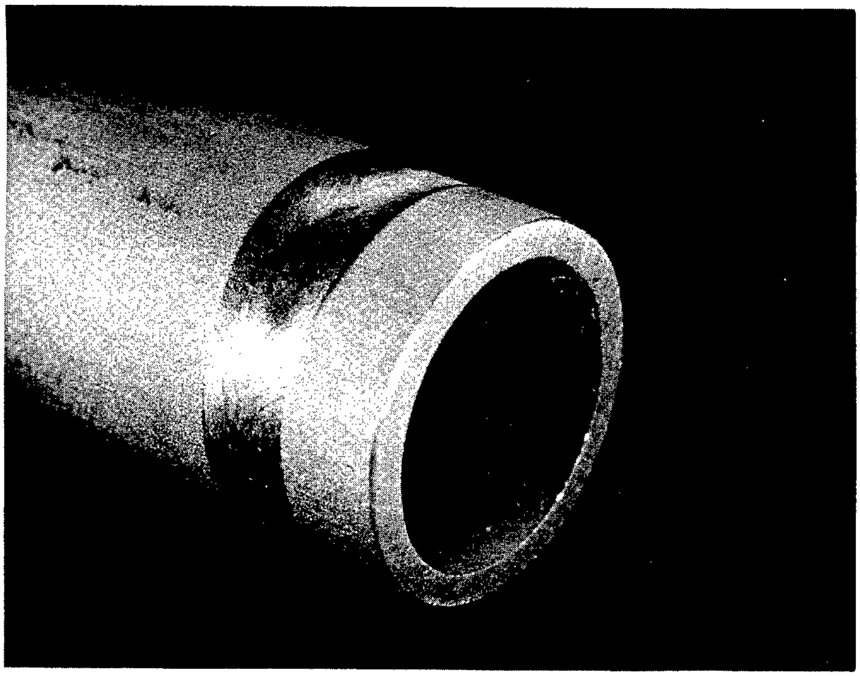

Figure 8 - Butt Weld in B-66 Tubing

TABLE III Phase II - T-111 Extrusion Billets

Billet No.	Weight (lbs)	Vickers Hardness (DPH)
T-111-II-1	37	242
T-111-II-2	38	224
T-111-II-3	37	228
T-111-II-4 (a)	38	240
Total	150	

(a) Eight pounds of material were subsequently lost during remachining due to extrusion malfunction.

Extrusion of the Phase II B-66 and T-111 billets to a nominal 2 inch O.D. x 0.25 inch wall tube blank was accomplished at the facilities of Nuclear Metals Inc. on July 23 and 24, 1963. The results of the tube blank extrusion are summarized in Table IV. The mild steel clad extrusion billets were heated in an argon purged retort for a minimum of four hours prior to extrusion. For the B-66-II-1 extrusion, the mandrel was heated to 900°F. However, during the extrusion the mandrel hung up in the billet and was pulled through the die. The mandrel was not heated for the balance of the extrusions. T-111-4 stalled the press for no apparent reason. This billet was reprocessed remachined at Nuclear Metals Inc. and then returned to WANL for annealing. The billet was annealed for one hour at 1600°C (2900°F). The annealed hardness was 225 DPH. Eight pounds were lost from T-111-4 during the re-machining operation, thus reducing the total starting weight of the Phase II T-111 extrusion billets to 142 pounds. T-111-4 was re-extruded successfully on September 9, 1963. The mild steel cladding was removed from the extrusions by pickling. The B-66 and T-111 tube blank extrusions are shown in Figures 9 and 10. Moderate to severe rippling was observed on all the extrusions with the exception of T-111-4 which was free of ripples. B-66-II-2 exhibited generally severe rippling and cir-

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TABLE IV Phase II Tube Blank Extrusion[(a)]

Extrusion (sequence)	Ram Speed (in. per min.)	Extrusion Force (tons)	Extrusion Constant (ksi)[(b)]	Remarks
B-66-II-1 (1)	300	1300	81.7	Mandrel failure, moderate rippling
B-66-II-2 (5)	500	1270	80.0	Circumferential cracks, heavy rippling
B-66-II-3 (6)	250	1300	81.7	Moderate rippling
B-66-II-4 (7)	320	1280	80.5	Moderate rippling
T-111-II-1 (2)	675	1360	85.5	Moderate rippling, 3" section hung up on mandrel
T-111-II-2 (3)	830	1240	78.0	Moderate rippling
T-111-II-3 (4)	875	1210	76.0	Moderate rippling
T-111-II-4[(c)] (8)	1000	1240	78.0	Good surface

Notes:

(a) Liner - 4.55" diameter @ 900°F
Die - 2.167" diameter with 90° included angle, @ 900°F
Mandrel - 1.411" diameter (Floating type) @ 900°F for B-66-II-1, room temperature for all others
Cut off - Mild steel @ 1200°F
Reduction Ratio - 7:1
Lubrication - Glass pad in die, mica on liner and mandrel
Billet heating - B-66 - 2200°F ±25°F (Minimum heating time,
T-111 - 2300°F ±50°F 4 hours)

(b) Calculated from $P = A_L K \ln R$ where A_L = cross-sectional area of liner, P = Extrusion Force, R = Reduction ratio

(c) Stalled on first attempt - no explanation.

Figure 9 - Phase II T-111 Tube Blank Extrusions

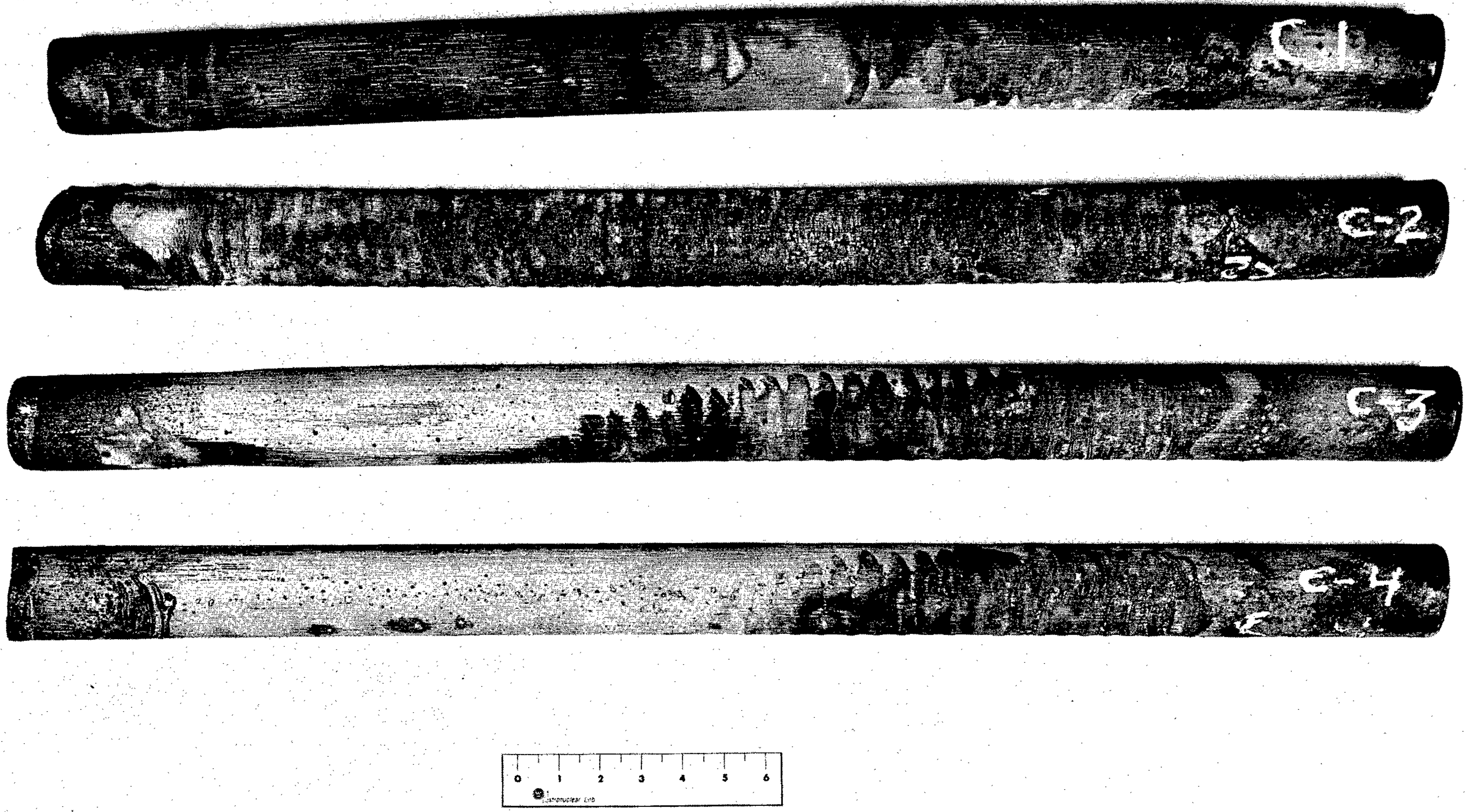

Figure 10 - Phase II B-66 Tube Blank Extrusions

cumferential cracking which was attributed to excessive extrusion speed. The rippling on the balance of B-66 and T-111 extrusions was confined to localized areas. Representative defective and satisfactory areas of the B-66 and T-111 extrusions are shown in Figures 11 and 12.

The limited number of extrusions made on this program prevented a detailed study of the tube blank extrusion process. Thus the parameters selected for the extrusions, while satisfactory, were not optimum.

The extrusions were cropped at the nose and tail, and a section was removed from the center of the extrusion. Dimensional measurements made at the nose, center, and tail location of the extrusions are summarized in Table V. The measurements are the averages of three readings taken 120° apart. To obtain a starting tube blank size of 2 inch O.D. x 0.250 inch wall will necessitate conditioning losses from the I.D. and O.D. totalling approximately 30%.

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The metallurgical condition of the tube blank extrusions was similar to that of the Phase I solid extrusions. This was expected since both the solid and tubular billets were given the same reduction under essentially identical extrusion conditions. Table VI contains the results of the metallurgical evaluation of the Phase II billets. The results verified the findings under Phase I and are reiterated below:

1. B-66 will be given the initial tube reduction in the as-extruded condition.

2. T-111 will require annealing prior to the initial tube reducing pass.

3. The mild steel cladding afforded excellent protection during the extrusion process.

Photomicrographs from the as-extruded B-66 and T-111 are shown in Figures

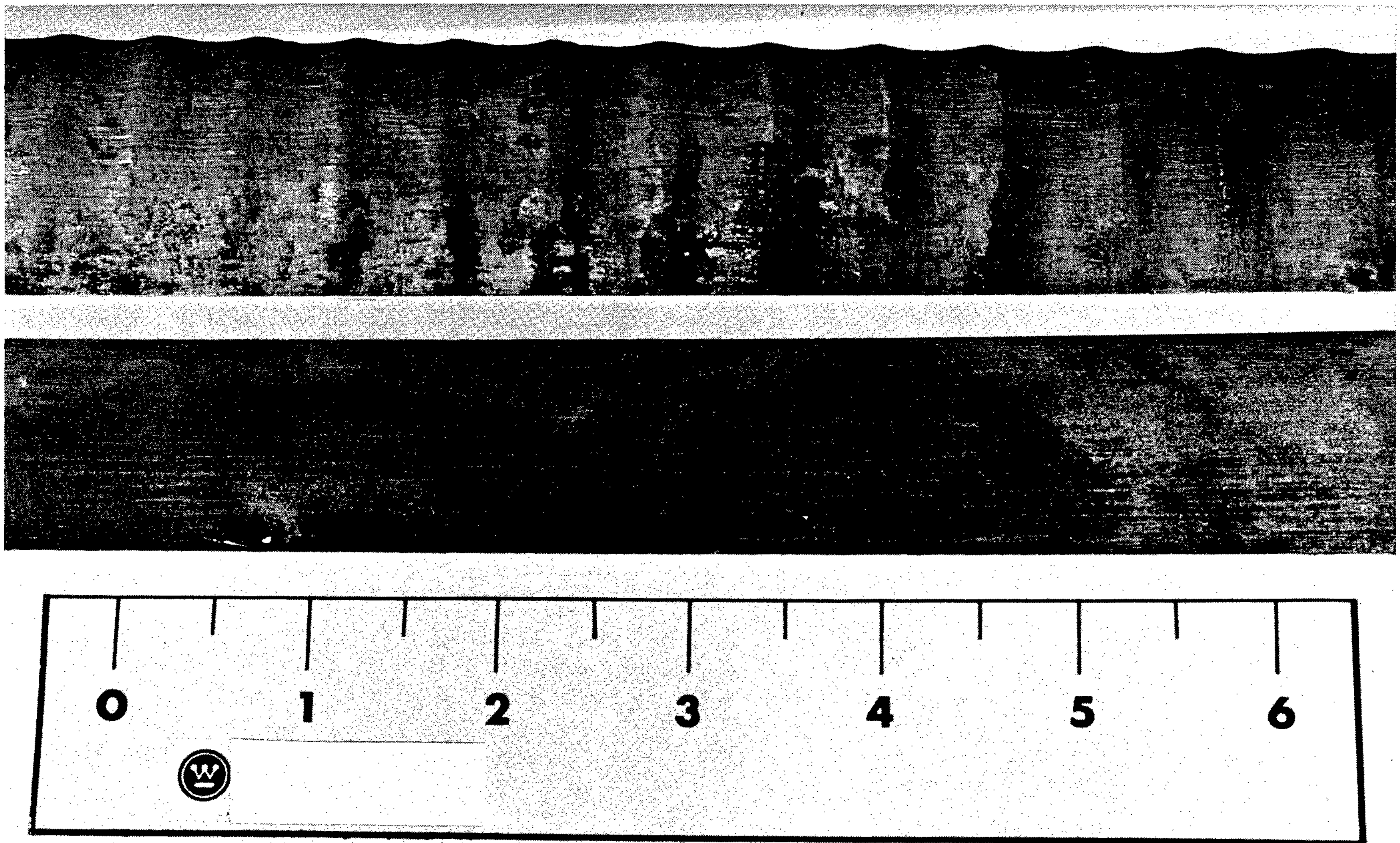

Figure 11 - T-111 As-Extruded Surfaces

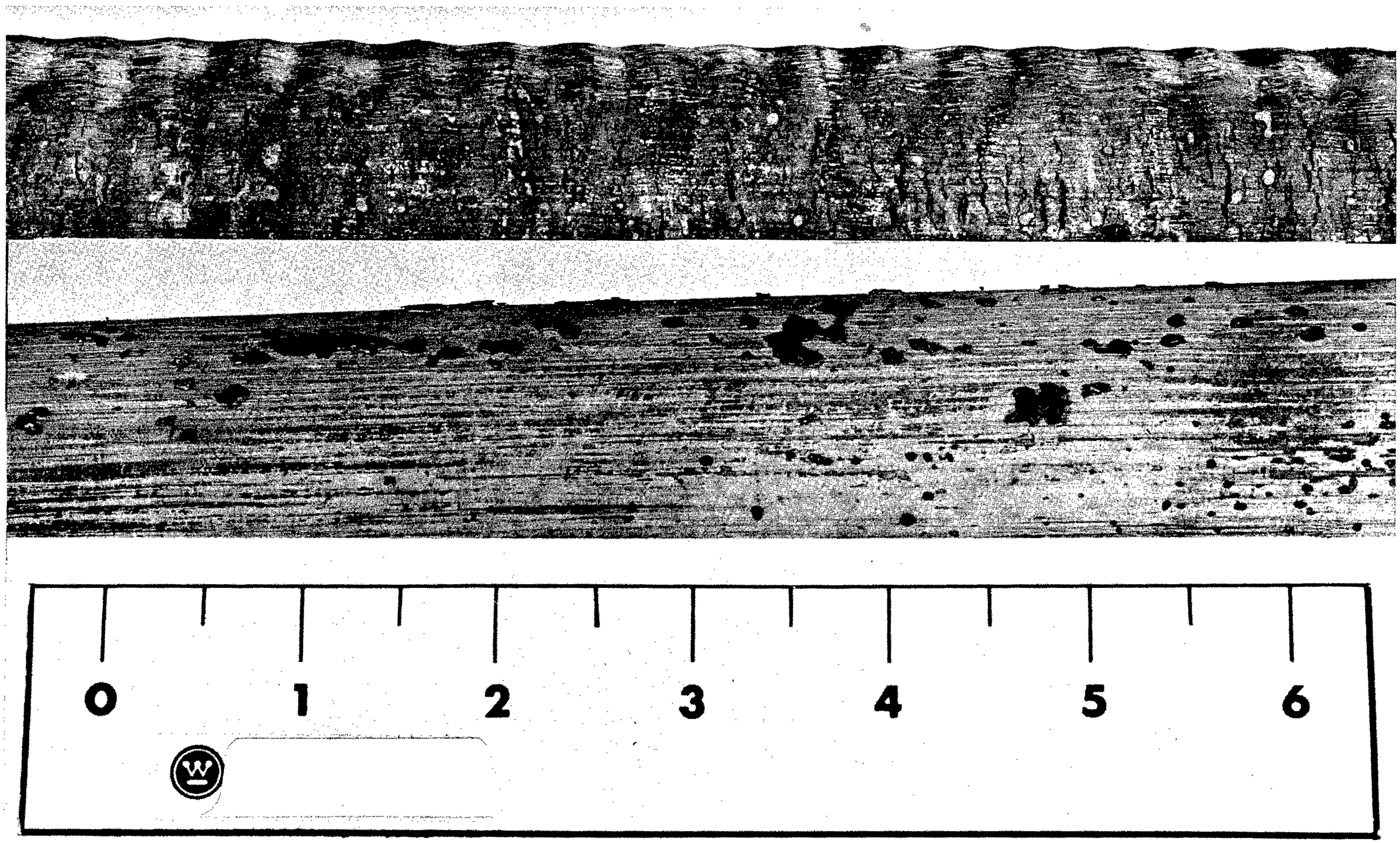

Figure 12 - B-66 As-Extruded Surfaces

TABLE V   Dimensions of the Phase II Tube Blank Extrusions

Billet	O.D.	I.D.	Wall	Remarks
B-66-1				
Nose	2.107	1.450	0.328	0.304-0.354 wall variation
Center	2.115	1.425	0.345	Out of round
Tail	2.140	1.425	0.345	Mandrel lodged in billet
B-66-2				
Nose	2.155	1.425	0.365	Out of round
Center	2.140	1.425	0.357	0.325-0.360 wall variation
Tail	2.160	1.395	0.380	0.350-0.380 wall variation
B-66-3				
Nose	2.160	1.455	0.352	Out of round
Center	2.155	1.465	0.345	Out of round
Tail	2.195	1.433	0.381	Out of round
B-66-4				
Nose	2.145	1.450	0.347	--------
Center	2.120	1.418	0.357	--------
Tail	2.170	1.390	0.390	--------
T-111-1				
Nose	2.088	1.445	0.321	--------
Center	2.125	1.425	0.350	0.345-0.375 wall variation
Tail	2.145	1.446	0.349	--------
T-111-2				
Nose	2.110	1.452	0.329	0.308-0.360 wall variation
Center	2.109	1.400	0.354	0.335-0.355 wall variation
Tail	2.140	1.405	0.367	--------
T-111-3				
Nose	2.120	1.450	0.335	--------
Center	2.112	1.420	0.346	0.337-0.355 wall variation
Tail	2.140	1.405	0.367	--------
T-111-4				
Nose	2.070	1.485	0.293	--------
Center	2.090	1.485	0.293	--------
Tail	2.130	1.470	0.330	--------

TABLE VI Effect of One Hour Annealing Temperatures on Hardness of Phase II B-66 and T-111 Extrusions

	As Extruded		Annealing Temp. °C/°F			
	Transverse	Longitudinal	1050/1900	1100/2000	1150/2100	1200/2200
B-66						
Nose	236	235	241	234	229	226
Center	230	228	234	227	226	225
Tail	233	232	234	229	227	228

	As Extruded		Annealing Temp. °C/°F			
	Transverse	Longitudinal	1350/2450	1400/2550	1450/2650	1500/2700
T-111						
Nose	297	297	249	235	235	231
Center	293	293	241	237	237	235
Tail	301	295	242	236	236	232

13 and 14. The nose portion of the B-66 exhibited predominantly an elongated microstructure although the hardness measurements indicated recovery of annealed mechanical properties.

The O.D. of the B-66 tube blanks was conditioned by turning, and the I.D. by boring. The conditioned B-66 tube blanks are shown in Figure 15, and a summary of the yield from the extrusion billets is given in Table VII. Cb Dye penetrant

TABLE VII Yield of Conditioned B-66 Tube Blanks

Extrusion No.	Extrusion Weight lbs.	Conditioned Tube Blank Weight, lbs.	Yield per cent
1	17.4	10.00	57.5
2	17.2	9.75	56.7
3	17.3	11.30	65.3
4	17.5	12.60	72.0
Total	69.4	43.65	63.0

inspection did not show any O.D. defects. The ultrasonic indications were attributed to localized surface conditioning and it was concluded that there were no internal defects greater than the standard used.

It had been planned to condition the T-111 by a combination of pickling and grinding. However the excess metal on the O.D. and I.D. prevented this from being a satisfactory operation. Thus, the T-111 billets are being lathe conditioned. After conditioning, the T-111 billets will be annealed for 1 hour at 1500°C (2700°F).

D. <u>Tube Reducing</u>

The conditioned B-66 tube blanks were shipped to the Superior Tube Company

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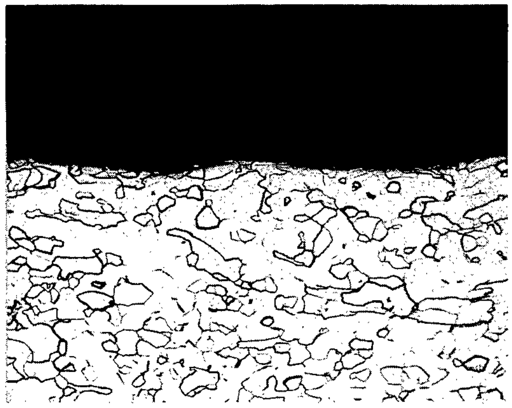
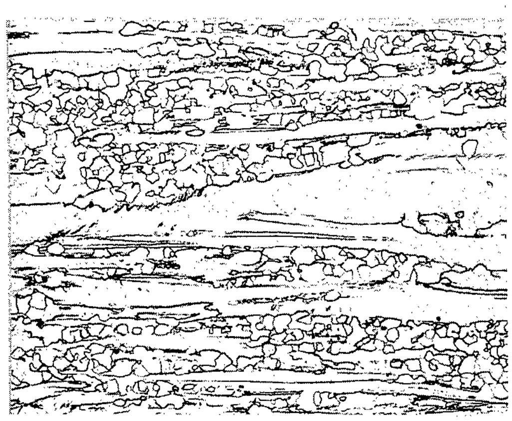
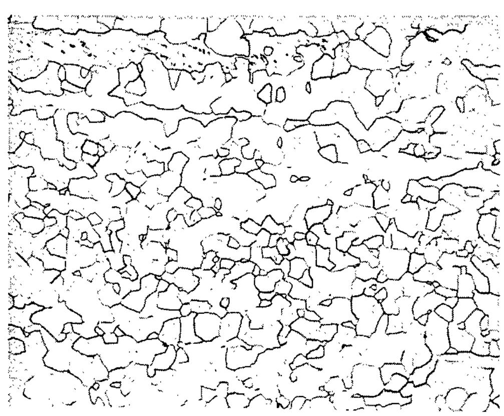


a. I.D. of B-66-II-1, 200X

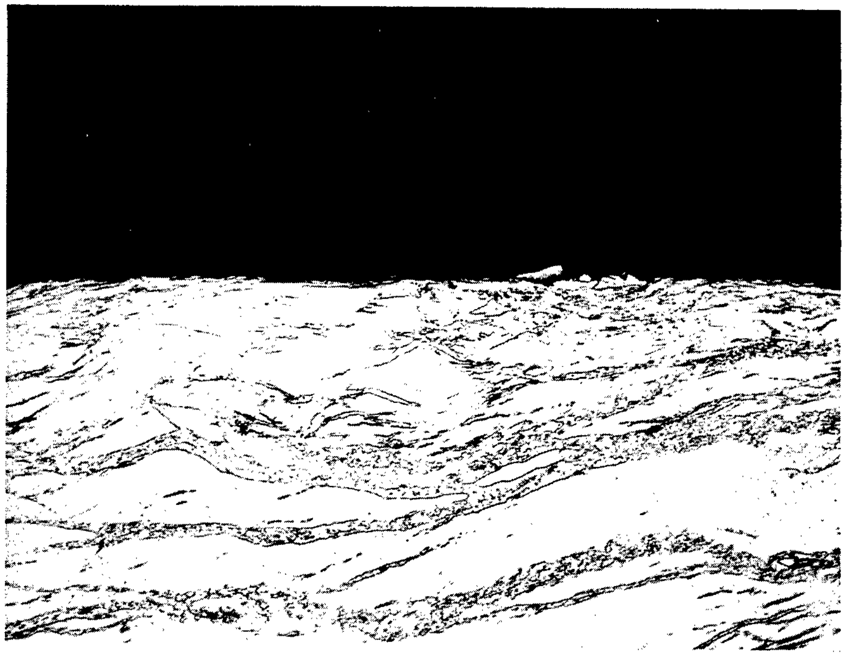


b. Longitudinal Section From B-66-II-1, 200X

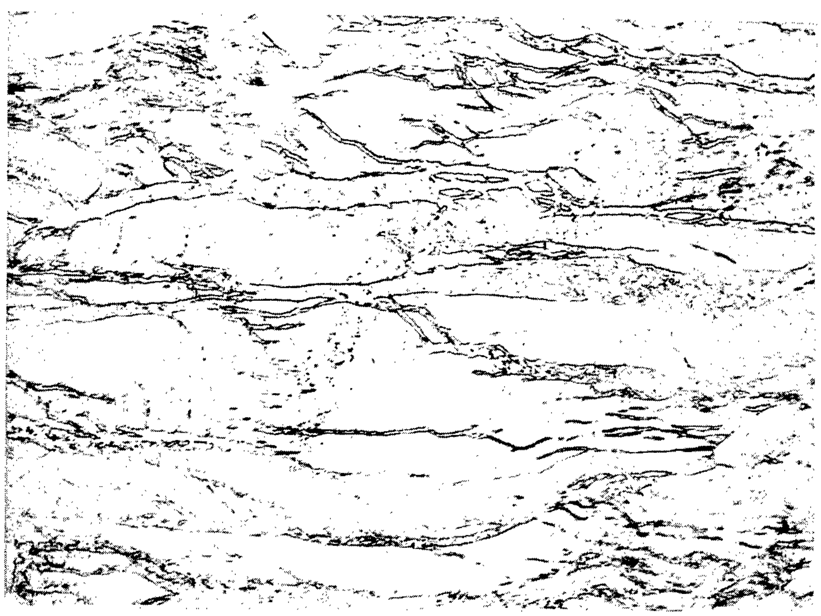


c. Longitudinal Section From B-66-II-2, 200X

Figure 13 - Microstructures of As-Extruded Phase II B-66



a. I.D. From T-111-II-1, 200X



b. Transverse Section From T-111-II-1, 200X

Figure 14 - Microstructures of As-Extruded Phase II T-111

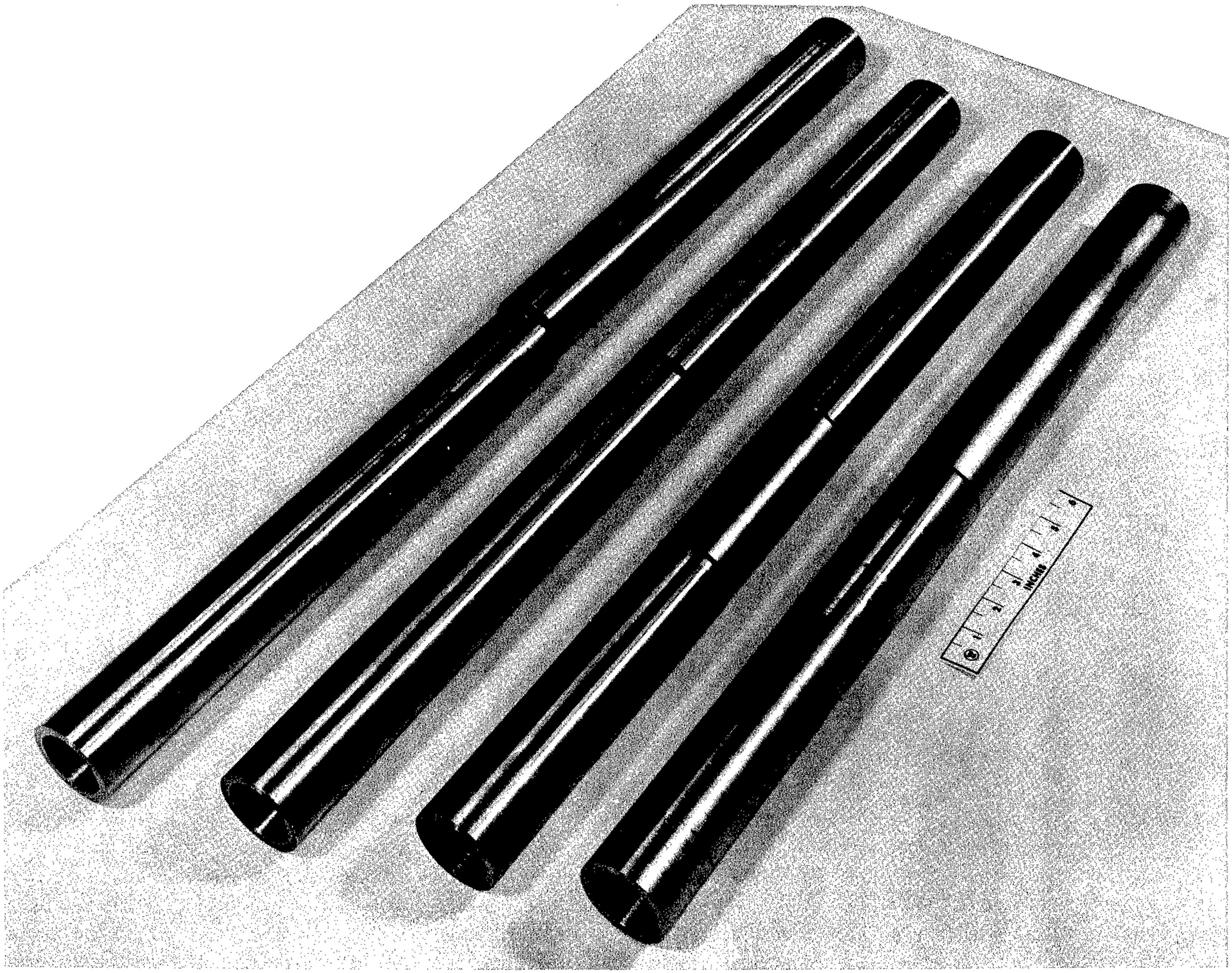


Figure 15 - B-66 Conditioned Tube Blanks

at Norristown, Pennsylvania. Plans were to tube reduce the B-66 at 500-600°F. A portion of a solid rod from the Phase I B-66 extrusion was swaged 75% at 120°C (250°F) indicating that tube reducing of the B-66 may not be limited to the 500°F minimum temperature initially selected.

A dry film lubricant, proprietary to the Superior Tube Company, was applied to the B-66 tube blanks. The billets were heated to 600°F in a resistance heated air furnace, then transferred to the mandrel of the tube rocker. A 4-5 foot length of mild steel pipe was used as a leader for the tube reducing operation. The initial tube reduction from 2 inch O.D. x 0.25 inch wall to a 1.375 inch O.D. x 0.156 inch wall was successful. The as-tube reduced blanks are shown in Figure 16. All nine tube blanks were reduced without a failure with the exception of one tube blank CIC which developed an inch long crack on one end.

The temperature measurement on the tube blanks during the tube reducing operation was extremely difficult to obtain. However, contact pyrometer measurements on tube blanks at the termination of the tube reducing operation indicated temperatures on the as-tube reduced B-66 from 280°F to as high as 400°F.

After tube reducing, the tubes were cleaned, stress-relieved for one hour at 1900°F in preparation for the second tube reducing pass to 0.875 inch O.D. x 0.109 inch wall. After the second tube reducing operation, the balance of the operations will be performed on the draw-bench. Room-temperature drawing Cb of B-66 will again be attempted utilizing different lubricants.

end

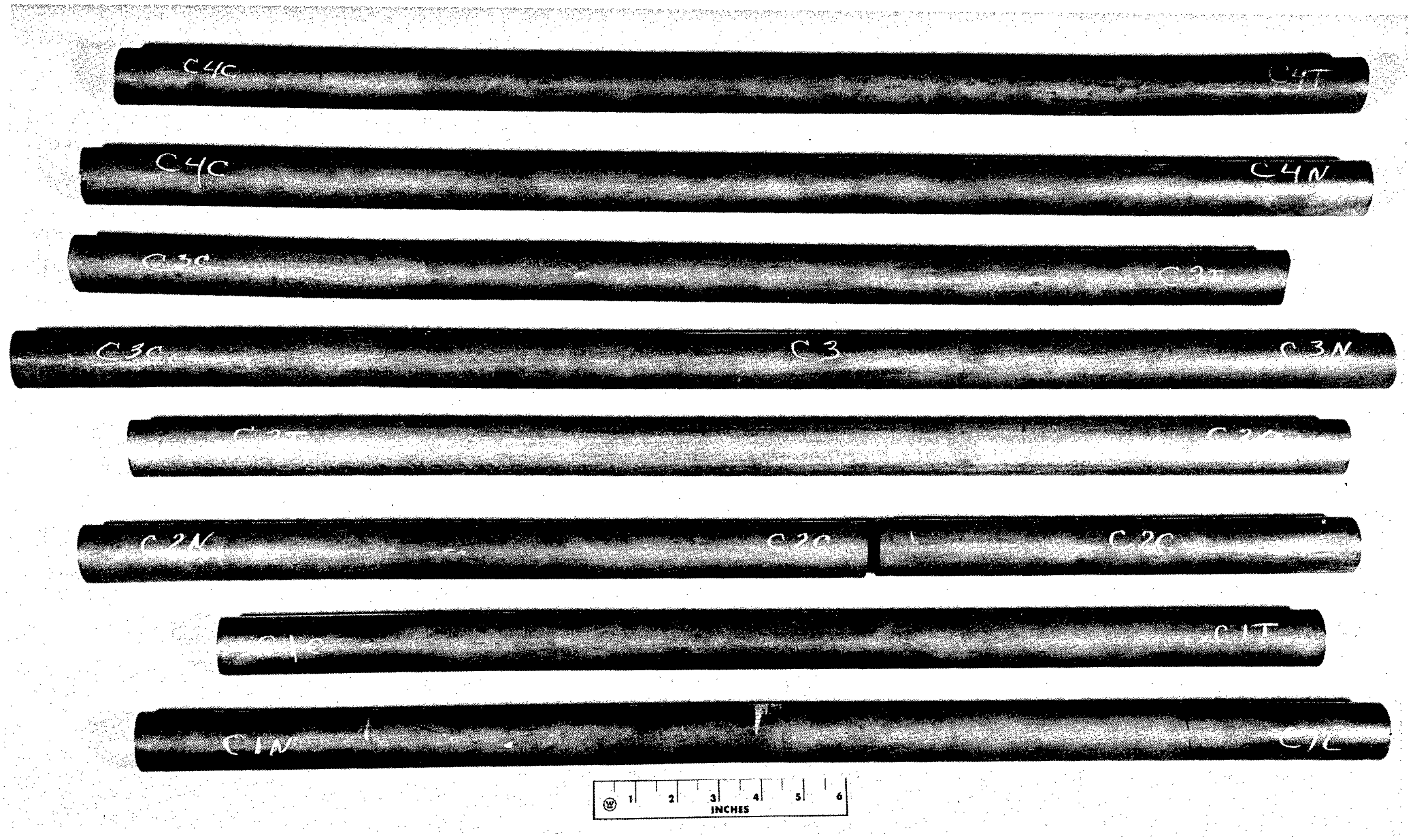


Figure 16 - B-66 Tube Blanks After First Tube Reducing Pass

III. FUTURE ACTIVITIES

During the final quarter, it is anticipated that the balance of the work will be completed and that the draft of final report will be prepared.

IV. REFERENCES

1. Buckman, R.W. - "Development of High Strength Columbium and Tantalum Alloy Tubing", 2nd Quarterly Progress Report, WANL-PR-(N)-002

2. Buckman, R.W. - "Development of High Strength Columbium and Tantalum Alloy Tubing", 1st Quarterly Progress Report, WANL-PR-(N)-001